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SYNTHESIS OF HYBRID COMPOSITES OF POLYSACCHARIDES BASED ON METHYLTRIMETHOXYSIANE

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Abstract: Hybrid composites of water-soluble acetyl cellulose and sodium carboxymethyl cellulose were synthesized using the sol-gel method. Their Fourier-IR spectroscopy and thermal stability were studied in the temperature range of 10-900°C.

Keywords: methyltrimethoxysilane (MTMS), water-soluble acetyl cellulose (WSAS), sodium carboxymethylcellulose (Na-CMC), sol-gel method, hydrite composite, Fure-IR spectroscopy, TGA-DTA analysis.

Introduction. Today, the demand for materials based on polysaccharides with a specific structure and their hybrid compositions is increasing due to their high sorption properties. By changing the pore structure of hybrid composites, it is possible to further expand the fields of application [1]. In addition, WSAS are widely used as stabilizers in the pharmaceutical industry. Thermodynamic properties of water-soluble acetyl cellulose and collagen-based composites [2], sorption capabilities are detailed [3].

Water-soluble polyelectrolytes play a crucial role as stabilizers and ingredients in a wide range of formulated products. Na-CMC is a polyelectrolyte widely used as a rheological modifier [4]. Dilution of Na-CMC solution with a monovalent salt solution results in the formation of a polyelectrolyte [5].

Calculations of the thermodynamic parameters of complex formation in dilute solutions, as well as physicochemical studies, show that the formation of the interpolymer complex occurs due to the cooperative interaction of Na-CMC carboxylate anions with protonated nitrogens of proteins [6]. In recent years, interest in the physicochemical properties of water-soluble cellulose acetate, which has been widely used in many industries, has been growing. In this regard, a number of studies have calculated the thermodynamic parameters of the polymer-solvent interaction by studying the water sorption properties of the same polymer substance and the composite mixtures based on it [7-10].

Experimental part. At the initial stage of the synthesis process, a solution of WSAS and MTMS is prepared. The resulting solutions were stirred with 0.1 M NH₄OH in the presence of ethanol at 50°C for 30 minutes. The sample formed in the second stage is dried at room temperature for 24 hours, and in a drying oven at 50-55°C until its mass does not change. Synthesis of the hybrid composition of Na-CMC was carried out in the same way.

The synthesized hybrid compositions were conditionally designated A1, A2, B1, and B2 according to the ratio of WSAS and Na-CMC.

Hybrid composites were synthesized, and the molecular masses of WSAS and Na-CMC were compared using viscosimetric and gel chromatography methods. Gel

chromatography (exclusive liquid chromatography) was performed on an Agilent 1260 Infinity liquid chromatograph using a PL Aquagel OH Mixed chromatographic column with a length of 25 cm and an internal diameter of 0.8 cm. When a 0.1 M solution of sodium nitrate was used as the eluent, the volumetric flow rate of the eluent was 0.8 ml/min, and the gel chromatograms were recorded using a differential refractometer (RID10A, Shimadzu) detector, the molecular masses of WSAS and Na-CMC were 100,000 and 80,000, respectively. The analysis results revealed that the values obtained experimentally using the viscometric method differ from each other. Since the viscometric method is a relative method, and gel chromatography is a highly accurate method for determining molecular mass, Figure 1 shows the molecular masses of WSAS and Na-CMC determined by gel chromatography.

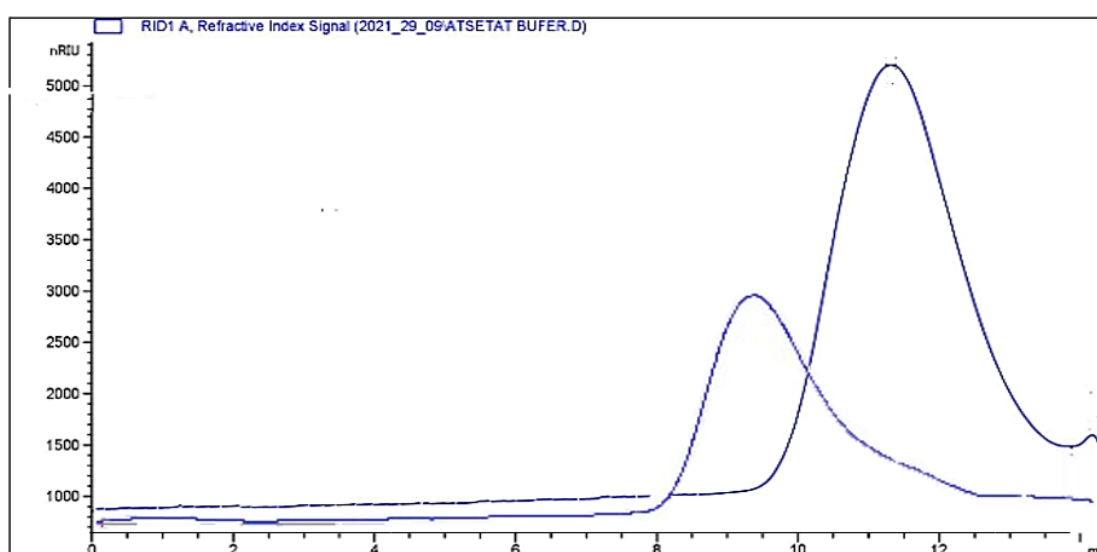


Figure 1. Molecular mass determination graph of WSAS (a) and Na-CMC(b) by gel chromatography method

The structure of the resulting silica hybrid composites was analyzed using IR-Fure spectroscopy. The infrared spectra of the samples A1 and A2 (1:1 and 1:5 ratio) hybrid composition based on WSAS of kremnezem are presented in Figures 2 and 3.

From the vibration frequencies formed in the samples, we can see that the deformation vibrations of the O – H bond in the adsorbed water molecule occur in the 3379-3382 cm⁻¹ region, the vibrations of the CH₃ hydrophobic group in the 2970-2968-2917 cm⁻¹ region, the valence vibrations of the (CH₃)C=O acetyl groups after oxidation at 1727-1729 cm⁻¹, the valence vibrations of the C = C bond at 1639-1641 cm⁻¹, and the valence vibrations of the Si-CH₃ bond in the 1268-1270-764 cm⁻¹ region.

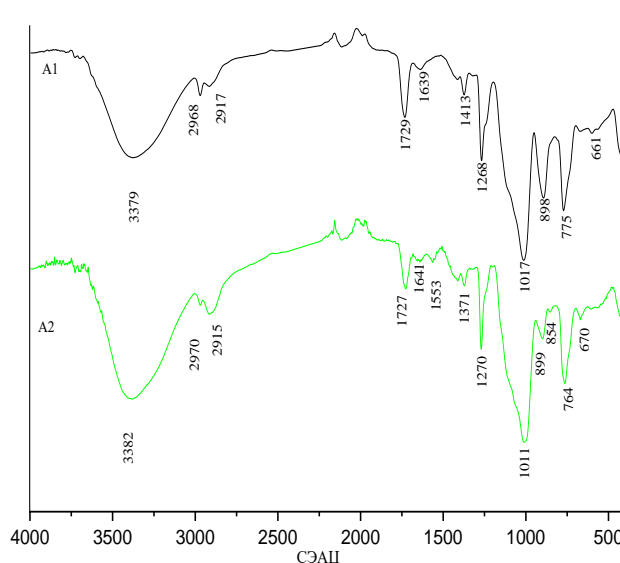


Figure 2. Analysis of IR spectroscopy results for silica composites obtained with WSAS

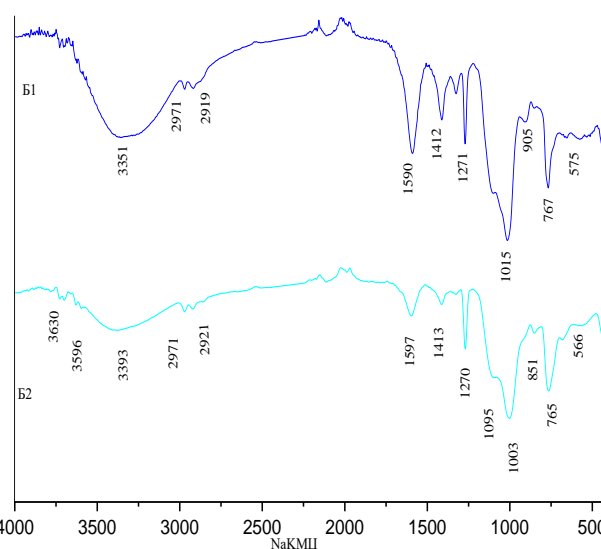


Figure 3. Analysis of IR spectroscopy results for silica composites obtained with Na-CMC

In addition, we can see that the A2 sample of the WSAS hybrid composite exhibits deformation vibrations of the N-H and C-C bonds in the region of 1553 cm^{-1} . This indicates that the synthesis process of the tebra in this area was carried out in an ammonia environment and that a small amount of ammonia remained during the drying process. From the results of this spectroscopic analysis, we can see that the spectra of both samples coincided with each other. Only the intensity of the formation of functional groups in the spectrum differs slightly depending on the ratio of the components. The similarity of the IR spectra of each sample indicates that they have a similar chemical structure.

When the formation of hybrid composites B1 and B2 of silica synthesized by the same method with Na-CMC (1:1 and 1:5) was carried out by IR-Fourier spectroscopy, vibrations in the O-H, C-H bonds were formed in regions similar to the above example. In the region of 1412-1414 cm^{-1} , Si-CH₃ deformation vibration and in the region of 1271-1270 cm^{-1} are showing valence vibration frequencies. While the vibrations in the 1015-1095 cm^{-1} region are associated with symmetric and asymmetric valence vibrations of Si-O-Si bonds, we can assume that the 905 cm^{-1} region is due to deformation vibrations of C-H.

Spectroscopic analysis of hybrid compositions shows that both samples form a mutual chemical bond. The thermal stability of the synthesized compositions was studied.

Thermal analysis was carried out in the range of 24-900°C on a "Thermal Analyzer KL-JS-1000A" derivatograph at a heating rate of 5 g/min and a gas flow of 1.50 ml/min. It was found that the initial weight of the silica composite obtained by WSAS decreased by 10% when heated to 74.5°C, i.e. the sample weight decreased from 14.9 mg to 13.41

mg. This mass loss is due to the release of water and volatile organic compounds from the sample.

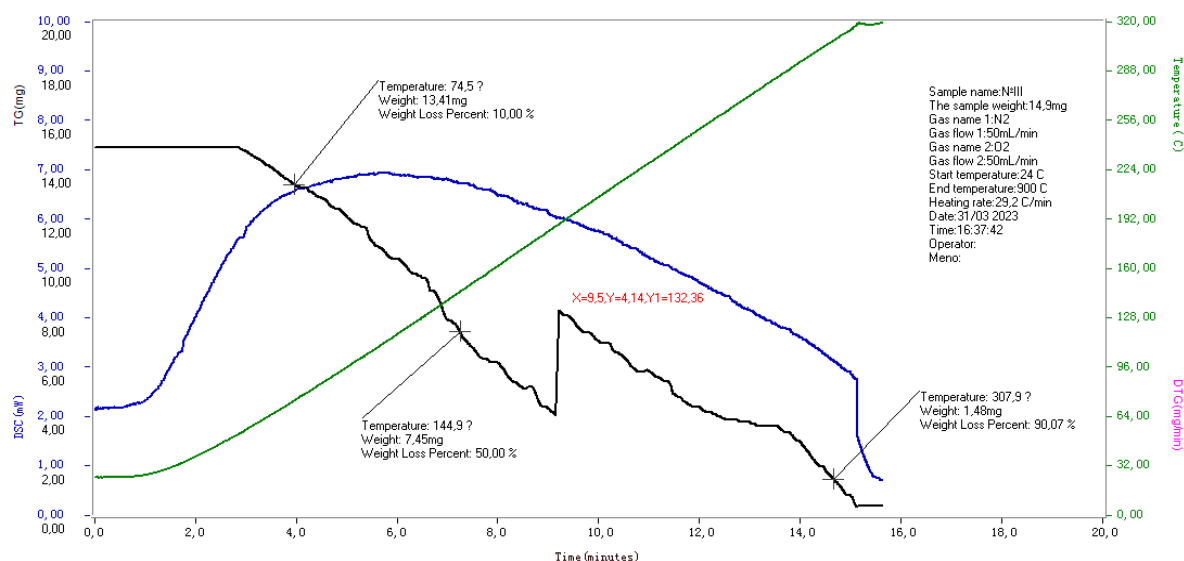


Figure 4. Thermal analysis of A2 hybrid composite obtained by **WSAS** of silica

When the temperature reaches 144.9°C, the mass decreases by 50%, as can be seen from the thermogravimetric curves in Figure 4. This indicates the release of -OH groups and the cleavage of acetyl bonds in the hybrid composite. When the temperature rises to 307.9 °C, the thermal decomposition of water-soluble acetyl cellulose in the hybrid composite decreases by 90%, indicating the presence of a 100% reduction in the mass of the material. We can conclude that the remaining mass is SiO₂.

Thermal analysis of the hybrid composite obtained with sodium carboxymethyl cellulose was also carried out in the same way.

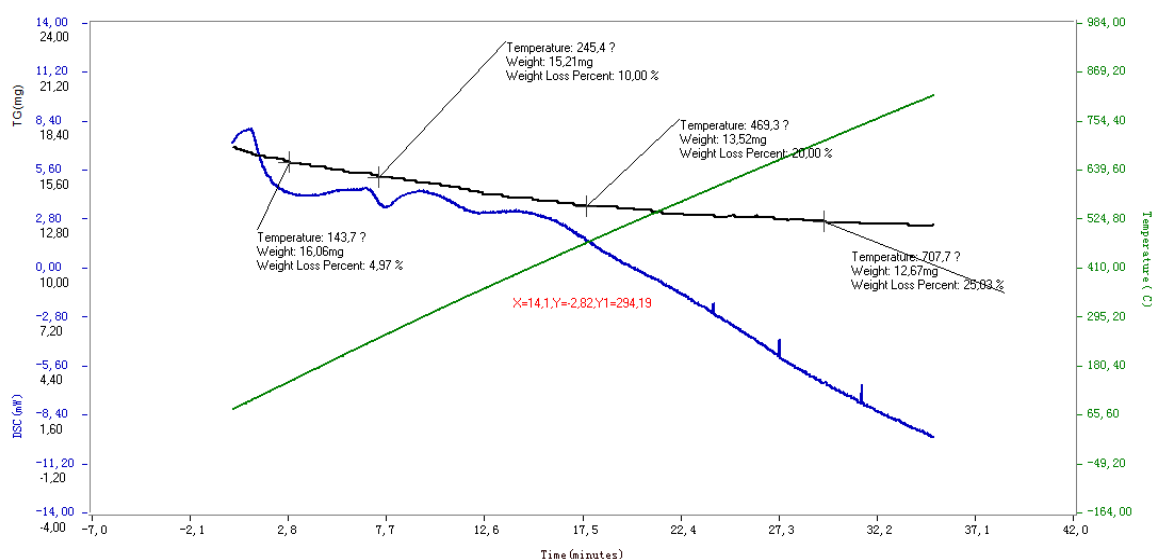


Figure 5. Thermal analysis of the B2 hybrid composite obtained with Na-CMC silica

However, we can see from Figure 5 that at a temperature of 143.7°C, the sample lost 4.97% of its mass. This is related to thermal sublimation. We can see that the mass of the substance decreases by 10% when it reaches 245.4°C. This mass loss is due to the release of OH groups. It was found that in the range of 245.4 - 469.3°C, the 20% decrease in the mass of the hybrid composite was due to the cleavage of carboxyl groups in carboxymethyl cellulose and the release of the methyl group, while when the temperature increased to 707.7°C, the mass of the material decreased by 25.93%. This weight loss is due to the oxidation of the hybrid composite and the degradation of carboxymethylcellulose. We can assume that the remaining 39.1% of the mass belongs to silica.

The resulting hybrid composites have a highly porous structure, and the porosity parameters of the Na-CMC hybrid composite were studied using isothermal adsorption of water and benzene vapors. From the isotherms presented in Figure 6, it can be seen that the synthesized hybrid composite absorbed more water vapor than benzene vapor, which confirms that the sample has a high hydrophilic property.

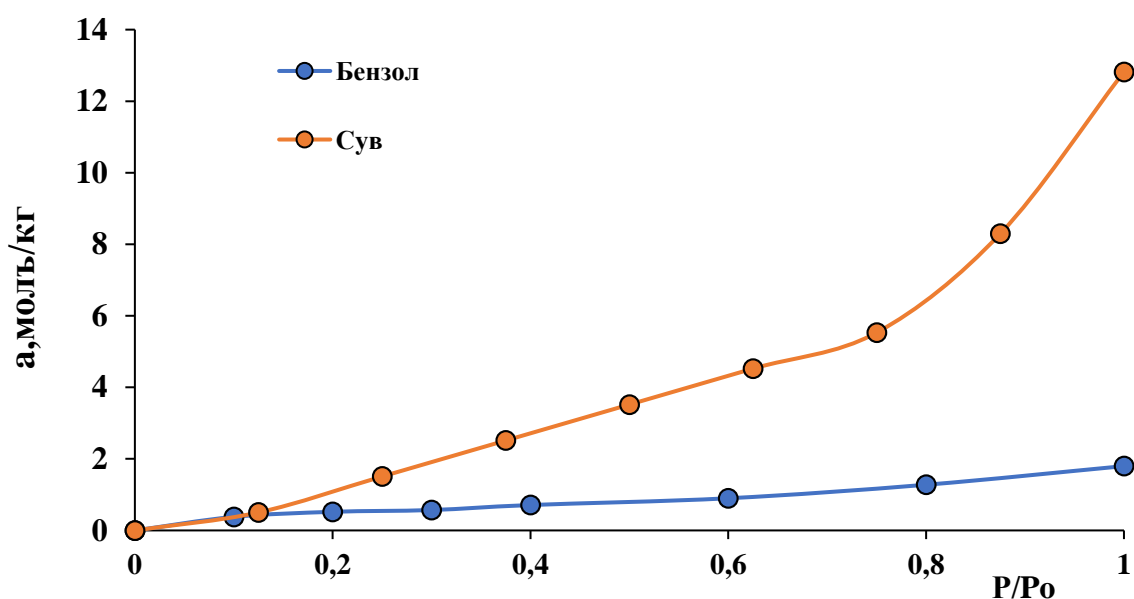


Figure 6. Porosity structure parameters calculated based on water and benzene vapor adsorption and desorption of the Na-CMC hybrid composite.

Based on the isotherms of water and benzene vapors, the pore structure of the samples (a_m – monomolecular layer capacity, S_{sol} – relative surface area, W_o – micropore volume, V_T – saturation volume, $W_{mez.}$ – mesopore volume) and thermodynamic parameters (ΔG_m – average Gibbs energy of mixing and ΔG_i – values of Gibbs energy) were determined (Table 1).

Table 1. Pore structure and thermodynamic parameters of WSAS and Na-CMC in water and benzene vapors

Porous structure and thermodynamic parameters	Water vapors according to	On benzene vapors
$a_m, \text{моль/кг}$	1,233	0,401
$S_{\text{сол}}, \text{м}^2/\text{г}$	80,19	96,36
$W_0, \text{см}^3/\text{г}$	0,11622	0,09804
V_T	0,23076	0,15936
W_{me}	0,11	0,06
$r_{\text{урта}}, \text{\AA}$	57,6	33,1
$-\Delta g^m, \text{Дж/г}$	9,60679	2,89085
$-\Delta G_i, \text{Дж/г}$	12,5	3,1

The results presented in the table show that the conclusion that the components of the composites are compatible in an aqueous environment is confirmed by the results of calculating the thermodynamic functions: The negative values of the thermodynamic functions for water sorption are much larger than those calculated from the benzene isotherms. Composites have higher specific surface area values for water vapor sorption, mean free energy of mixing, and more negative Gibbs energy. High negative values of Gibbs energy indicate that the sorption process is spontaneous.

Conclusions

1. The isotherms of the B2 hybrid composite obtained on the basis of Na-CMC for water and benzene vapors were compared. It was found that the hybrid composition has a high water vapor sorption property. Porous structure values are higher in terms of water vapor sorption, mean free energy of mixing and Gibbs energy are more negative, indicating hydrophilicity of the composition.

2. From the thermal analysis of the synthesized hybrid composites, it was found that the thermal stability of the B2 composite is higher than that of the A2 sample.

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